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International Atomic Energy Agency
Agence internationale de l'énergie atomique
Международное агентство по атомной энергии
Organismo Internacional de Energía Atómica

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Ms. B. Hoffheins

ISPO Liaison Officer
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in Vienna (UNVIE)
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RECEIVED UNVIE-ISPO
DATE: AUG 1 1 2009

2009-08-04

Subject: Task Proposal 09/OAT-02

Dear Ms. Hoffheins,

With reference to the U.S. Support Programme, I am pleased to provide the following new Task Proposal:

09/OAT-02 Analysis of HALW-samples containing particles

This task is part of the Destructive Analysis of Nuclear Materials for Safeguards Project. The project manager is Mr. R. Lafolie.

Please inform me at your earliest convenience whether the content of the proposal is acceptable to the U.S. Support Programme or if you would like to introduce any amendments. In your reply, please also provide the name(s) of the U.S. contact person(s) responsible for the task.

Upon acceptance of the proposal, the IAEA will transmit the Task Outline to you indicating the name of the IAEA task officer. Our understanding is that the U.S. Support Programme and the IAEA task officer will then establish a detailed task workplan.

I would also like to express the appreciation of the IAEA for the valuable contribution provided by the U.S. Support Programme to the IAEA's safeguards efforts.

Yours sincerely,

Mark Pickrell
Acting Director
Division of Technical Support

Enclosure

SP-1
TASK PROPOSAL PART

1. Task Proposal

1.1 Task Proposal ID: 09/OAT-002 **Date received in SPA:** 2009-07-06

1.2 Task Title: Analysis of HALW-samples containing particles

1.3 Requester / Division / Section: Mayorov,V / SGOA / OAT

1.4 Is this a CFE task? No

1.5 Task Category: A

1.6 Is this a joint task for MSSPs? No

1.7 Is multiple acceptance required? No

If 1.6 or 1.7 is yes, indicate the reason:

2. Project

2.1 Project ID: SGTS-006 **Project Type:**

2.2 Project Title: Destructive Analysis of Nuclear Materials for Safeguards

2.3 Project Manager / Division / Section: Lafolie / SGTS / TTS

3. Safeguards Requirement Identification

3.1 What is needed, why and when:

What is needed :
To develop a method for determining Pu in HALW-samples received at the On-Site Laboratory at the Rokkasho reprocessing plant (OSL-RRP), which contain considerable amount of solid (metallic) particles, preferably a non-destructive assay (NDA) method.
The available equipment for the determination of Pu after sample pre-treatment is HKED as NDA method. Spectrophotometry of Pu(VI) as DA method is currently applied at OSL for determining Pu in the supernatant solution with large uncertainty due to residual particles, while Pu adsorbed at the particles cannot be determined by this way.

Why :
As part of the Safeguards Approach of the Rokkasho Reprocessing Plant (RRP), samples from High-Active Liquid Waste (HALW) which might contain solid particles and Fines streams are sent to the OSL. About 20 samples per year of this type are expected to be analyzed at the OSL. For the purpose of safeguards, the total Pu in such a sample is of interest, consisting of dissolved Pu and possibly Pu adsorbed and/or entrapped by the particles.
The concentration of Pu in these samples is low (< 1 g/L) and falls into the XRF-range of the HKED instrument inside the hot cell (HC) line at OSL (taking into account that software developments for XRF analysis are expected to lower the detection limits at OSL). However, OSL currently cannot reliably analyze these samples by XRF because homogenization of sample content by stirring is impossible in the HKED sample changer (which is covered by a neutron detector for Cm-244 assay). Without stirring, the particles settle and disappear out of the X-ray beam.
OSL currently determines the dissolved Pu in the supernatant solution after decantation by Pu(VI) spectrophotometry. Diluted acid is added to the sample to accelerate the settling of the particles. Decantation is, however incomplete, which causes significant measurement errors. Filtering turned out to be difficult at OSL due to the particle size (although filtering is applied to such samples at a reprocessing plant). However, after filtering, the particles would require a separate treatment which makes such option cumbersome. A centrifuge is not installed in the HC line.

The particles do not dissolve in nitric acid up to acid concentrations up to 65wt% nor in mixtures of acids. By heating such samples in the presence of concentrated acid or acid mixtures, it is expected that most of the adsorbed/entrapped Pu dissolves (this method is applied in some reprocessing plants, using an autoclave). However, if these samples are heated, volatile fission products (FP's) are released. These FP's (e.g. Ru-106) pass through the filters and escape into the exhaust ventilation system of OSL, triggering radiation alarm and evacuation. Such alarm was triggered each time after acid fuming in a HC at OSL.

When :
Task to be completed for July 2010.

User requirements :
- To determine the concentration of Pu (cPu) in heterogeneous solutions at concentrations between 0.01 < cPu < 1 g/L
- To carry out the analysis in a hot cell, because the samples are highly radioactive (mainly gamma-, beta-radiation). The hot cells at OSL are filled with many equipment, therefore any new equipment must be as small as possible, require as little as possible auxiliary reagents and must be operated via manipulators.
- The sample solutions are acidic (nitric acid) to prevent the cations from hydrolysis/precipitation
- Liquid waste generated from any process must not contain any organic compound or material
- Any new equipment must be corrosion-proof
- No additional equipment (e.g. stirrer) can be installed around the HKED sample changer, because the space is occupied by the neutron detector for Cm-244 assay.
- OSL handles and stores such samples in polyethylene-jugs, from which aliquots are taken under weight-control if required; in XRF-analysis the polyetheylene-jug is measured directly without aliquoting
- Filtering the particle-containing samples turned out to be unsuccessful for the samples received at OSL.
- The density of such samples cannot be measured because the particles damage the density meter
- The particle-containing samples represent only about 5% of the total sample throughput, therefore any new method should have little impact on sample logistics (blocking space required for treating/transporting other samples) and on operating resources (time for pre-treatment, time for measurement)

Proposed task :
It is proposed to investigate which methods are in use or under development in other safeguards laboratories and in analytical divisions in reprocessing plants to analyze HALW sample (containing particle) and Fines on a routine basis.
One preferred method for OSL could be XRF-analysis in stand-alone mode, or analysis XRF spectra self-induced by the fission products in the sample, using the HKED-instrument after homogenizing the sample. A scenario for homogenizing the sample could be applying a gelator (currently not used at OSL). However, gelating a sample is a common practice in other analytical methods.
The development of a scrubber for volatile FP's could be considered as another option. In that case, an aliquot would undergo acid fuming with a scrubber activated above it to absorb the fumes and FP's. However, a scrubber would need to be (re)movable in order not to interfere with heating and treatment of other samples. Also, a pump for a scrubber is difficult to be installed.
Other possible feasible methods shall be investigated (passive gamma measurement or neutron counting).
The proposed task shall therefore identify current method in operations or proposed alternative as suggested above considering the specifications of the OSL. For this support, OSL IAEA staff would supply to the selected SP member, after clearance from NMCC, the details of the OSL Hot Cell design that could accommodate the suitable analytical technique.
Depending of the proposed technical options and after discussion with OSL-IAEA staff, testing can be made before implementation in inactive condition for demonstrating the handling performance in Hot Cell environment before active testing are conducted with representative solutions.

3.2 How will the task results be used and by whom:

The quality of the analytical results of safeguards laboratories will benefit from such a development.

3.3 Consequences if task is not performed:

If OSL cannot establish a suitable method, the operator's declarations for Pu transfers or inventories relevant to the Waste Treatment and Storage Material Balance Area can not be confirmed and the performances of the Operator's Measurement System can not be assessed
The total amount of Pu involved is small (in the hundred-Gramm-range for a tank), but the uncertainty of the currently applied method (spectrophotometry on supernatant solution after decantation) is large.

4. IAEA Proposed Work Outline

4.1 Major task stages with timing:

The following phases are proposed:

Phase 1:
- To identify all potential techniques available or under development.
For each one, scope of application, performance, precision, and limitations should be provided.
- To evaluate and select the preferred method based on the review received.

Phase 2:
- To define a Statement of Work with the selected partner including well defined responsibilities.
- To demonstrate the performance (functional test).
- To validate the method and confirm that it meets the user requirements.

Phase 3:
- Field test
- Fine tuning
- Implementation

4.2 Support Division(s) / Section(s): NAAL / SAL
SGTS / TTS

4.3 End User Division(s) / Section(s): SGOA / OAT

4.4 Estimated duration in months: 12

5. Safeguards Approval Process - not displayed

6. Acceptance by MSSP(s)

6.1 MSSP(s) to which the task is proposed:	Date accepted:	Agency Task ID:
EC		
FRA		
GER		
JPN		
UK		
USA		